11. *SRMs to Support Chemical Measurements in Industrial Applications*

H. Chen-Mayer, S.J. Choquette, W.R. Kelly, R.M. Lindstron, E.A. Mackey, J. Mann, S.A. Margolis, A.F. Marlow, L. O'Neal, P.L. Paul, J.R. Sieber, R.D. Vocke Jr., and L.J. Wood

Objective: To promote U.S. economic growth by working with industry to develop and apply measurements and standards.

Problem: The development of new technology and products, coupled with the evolution and advancements of analytical instrumentation used by industry, results in an ever-expanding need for standards. The Division balances its broad program in compositional and chemical standards among continuously produced standards (e.g. primary materials), renewals (previously certified materials), and new standards. The Division works with broad-based industrial groups to identify and produce new standards—which typically will require the development of new analytical methodology or refinement of existing methodology—research in analytical methods.

Approach: New standard activities reside in all functional areas and Groups of the Division. Each project has its own unique characteristics, but each also has common stages: an indication of need by some industry advocacy group; the development and definition of NIST capabilities; and, then, the application of these NIST capabilities in value assignment of a material available for broad distribution as an SRM.

Results and Future Plans: Five examples of ongoing projects are provided to illustrate the breadth of industrial measurement problems being addressed by the Division.

<u>SRM 2035/2035a Near Infrared Wavelength:</u> NIR spectroscopy is a technique widely used in process control for a variety of industries. Through interactions with two committees of ASTM the need for optical filter wavelength standards in this wavelength region was established. In addition, technical workshops and meetings were held with users and regulators to refine these needs. Several prototype materials were evaluated; a round robin to potential end users in the chemical, polymer, pharmaceutical, and analytical instrumentation industries were completed. Technical issues such as homogeneity, temperature and environmental stability, polarization and bandwidth effects required investigation. The first material has now been certified.

<u>SRM 861, Aerospace Superalloy:</u> The U.S. aerospace industry is a leading technology driver in R&D spending and the nation's leading net exporter of manufactured products. The aircraft industry and supporting foundries and their Consortium on Casting of Aerospace Alloys brought to NIST the need of the industry to produce turbine blades with sulfur concentrations near 1 mg/kg, a critical level between acceptable and unacceptable materials. Presently, the two established techniques used by industry to measure sulfur are discordant and both require standards for calibration and validation—standards that did not exist. The Division has investigated the extension of its primary isotope dilution mass spectrometric method to the low concentration required, accounting for the variability of the blank, the limiting source of uncertainty in this measurement. A material has been prepared to benchmark the need and NIST's measurement capability. Preliminary results indicate that the certified sulfur value will be an order of magnitude lower than any other standard.

<u>SRM 2721/2722 Moisture in Crude Oil:</u> The measurement of moisture in crude oils is important for the oil industry because of the large amount of crude oil that is recovered by a steam process and shipped saturated with water. ASTM methods existed for this measurement; however reference materials do not exist to benchmark the accuracy of these methods, or to gauge their robustness across materials and conditions. In preparation for certification, these methods have been critically assessed and sources of systematic bias have been enumerated. One result was the identification of a large negative bias for the recommended ASTM method D1533 if the oil is not completely dissolved or if the instrument is not properly calibrated. The development of a reliable method has resulted in moisture certification in two different crude oil materials at approximately 926 and 96 mg/kg. It is also intended to certify these two materials for their sulfur content, another chemical component that strongly affects crude oil value.

<u>Hydrogen in Titanium Alloys:</u> Hydrogen is one of the chief contributors to brittleness in metals; its measurement and control in certain alloys is critical,

especially for the aerospace and nuclear power industries. The principal method used in industry to measure hydrogen is calibrated with working standard materials. At one time NIST used this method and certified materials in co-operation with industry. However, NIST no longer supports either this technique or the mode of certification where no NIST measurements are included. The ASTM metals standards committee has steadfastly maintained the importance of benchmarking this measurement. Thus, a method has been developed that can produce metal standards of absolutely known hydrogen concentration. This method is based on the controlled reaction of hydrogen with titanium. Homogeneity is checked by neutron incoherent scattering and concentration is verified by cold-neutron prompt-gamma activation analysis. The first material has been prepared near the critical level of 100 mg/kg. In addition, new materials will be prepared at two bracketing levels.

SRM 1848 Lubricant Additive Package: The lubricants industry is estimated to be worth $5 billion in the United States. Although ASTM maintains a range of analytical methods for lubricants, none of the methods have bias statements, and there are no certified reference materials to verify the accuracy of analysis. Thus, a SRM certified for additive elements in passenger car motor oil was proposed to fill the need for traceable standards for this industry, and this standard is in the process of being certified in co-operation with industry laboratories. The candidate material was donated by one of the major U.S. producers, its homogeneity checked at NIST, and a round robin of major industry laboratories has been conducted. Quantitative procedures for XRF analysis have been implemented at NIST to benchmark established NIST and industry instrumental methods. At NIST, certification measurements for 8 elements, B, N, Mg, P, S, Cl, Ca, and Zn, by nuclear methods, XRF, atomic spectroscopy, and mass spectrometry are in progress. The goal is to achieve uncertainties for the NIST certified values of 0.5-1.0% to meet the industry's need (~3%).